IS 326-13 (2005): Methods of Sampling and Test for Natural and Synthetic Perfumery Materials, Part XIII: Determination of Cineole Content [PCD 18: Natural and Synthetic Fragrance Materials]

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IS 326 (Part 13) : 2005
ISO 1202 : 1981

भारतीय मानक

प्राकृतिक और संश्लेषित सुगन्ध सामग्री के नमूने लेने और परीक्षण की पद्धतियाँ

भाग 13 1,8-साइनओल अंश ज्ञात करना

(तीसरा पुनरीक्षण)

Indian Standard

METHODS OF SAMPLING AND TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

PART 13 DETERMINATION OF 1,8-CINEOLE CONTENT

(Third Revision)

ICS 71.100.60

BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

May 2005

Price Group 2

NATIONAL FOREWORD

This Indian Standard (Part 13) (Third Revision) which is identical with ISO 1202 : 1981 'Essential oils — Determination of 1,8-cineole content' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendations of the Natural and Synthetic Fragrance Materials Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The text of ISO Standard has been proposed to be approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to the following International Standard for which Indian Standard also exists. The corresponding Indian Standard, which is to be substituted in its place, is listed below along with its degree of equivalence for the edition indicated. However, that International Standard cross-referred in this adopted ISO Standard, which has subsequently been revised, position in respect of that latest ISO Standard has been given:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 212 : 1973 Essential oils — Sampling	IS 326 (Part 1) : 1984 Methods of sampling and test for natural and synthetic perfumery materials: Part 1 Sampling (*second revision*)	Equivalent

The Technical Committee responsible for the preparation of this standard has reviewed the provisions of the followiing International Standard and decided that this is acceptable for use in conjunction with this standard:

International Standard	*Title*
ISO 356 : 1996	Essential oils — Preparation of test samples

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHODS OF SAMPLING AND TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

PART 13 DETERMINATION OF 1,8-CINEOLE CONTENT

(Third Revision)

1 Scope and field of application

This International Standard specifies a method for the determination of the content of 1,8-cineole in essential oils, the principal constituents of which are 1,8-cineole and terpene hydrocarbons.

The method is also applicable to the essential oils of cajuput and niaouli, provided that an appropriate table is used; such a table will be included in the section entitled "Requirements" in the relevant International Standards dealing with these essential oils.

Parallel to this method, methods for the determination of 1,8-cineole in certain essential oils, by gas chromatography, will be the subject of future International Standards.

2 References

ISO 212, *Essential oils — Sampling.*

ISO 356, *Essential oils — Preparation of test sample.*

3 Principle

Measurement of the crystallization temperature of a mixture of essential oil and *o*-cresol. This temperature depends on the 1,8-cineole content of the essential oil.

4 Reagents

4.1 ***o*-Cresol,** purified, anhydrous, melting-point not less than 30,5 °C.

As this reagent is hygroscopic, it should be stored in small, well-stoppered bottles, or preferably in sealed flasks. These containers should also be protected from light.

When the *o*-cresol is not in the condition specified above, it is possible to purify it as follows :

Melt a quantity of *o*-cresol (analytical reagent grade), add 5 % of its mass of distilled water, and allow to crystallize at a temperature of 20 to 25 °C. Drain the crystals, and transfer them to a flask fitted with a fractionating column. Distil the first 10 % (*V*/*V*) and discard it. Replace the column by a similar one, but dry, and distil 80 % (*V*/*V*), the residue in the flask being discarded. Allow the main fraction to crystallize. If its melting point is still below 30,5 °C, repeat the distillation as before, as many times as is necessary to obtain a product having a melting point not less than 30,5 °C, which is colourless on melting.

4.2 1,8-Cineole, analytical reagent grade.

The purity of cineole shall be checked, for example, by measurement of the refractive index at 20 °C, which shall be between 1,455 0 and 1,460 0.

4.3 1,8-Cineole-*o*-cresol complex, prepared by mixing equimolecular proportions (in the ratio 154,24/108,13) of the cineole (4.2) and the *o*-cresol (4.1), and purified by crystallization from light petroleum (of analytical grade), of distillation range between 40 and 60 °C. The crystallization point of the complex shall not be below 55,2 °C.

5 Apparatus

5.1 Calibrated thermometers, mercury in glass, fulfilling the following requirements :

- length of bulb : 10 to 15 mm;
- diameter of bulb : 5 to 6 mm;
- graduation : 0,1 °C;
- calibration : 0,1 °C.

The set of thermometers used shall permit the measurement of any temperature between 20 and 60 °C.

5.2 Ordinary thermometer.

5.3 Test tube, about 20 mm diameter and 100 mm long.

5.4 Stout-walled test tube, about 30 mm diameter and 125 mm long.

5.5 Apparatus assembly for determination of freezing point. (See the figure, which is given as an example.)

It consists of a wide-mouthed jar or bottle of about 500 ml capacity, provided with a bored cork or rubber stopper into which the stout-walled test tube (5.4) is inserted. The test tube (5.3) is fitted into the stout walled test tube (5.4) by means of another bored cork or rubber stopper.

If necessary, the above-mentioned vessel may be filled with cold water for cooling prior to the preliminary test (see 7.2) and to the actual determination (see 7.3).

The thermometer (5.1) is inserted into the test tube (5.3) so that the centre of the mercury bulb is located at the centre of the liquid.

5.6 Water bath.

5.7 Agitator.

6 Sampling

See ISO 212.

7 Procedure

7.1 Preparation of test sample

See ISO 356.

7.2 Preliminary test

Weigh, to the nearest 0,001 g, 3 g of the freshly prepared test sample (see 7.1) in the test tube (5.3) carefully dried, and add 2,10 g of the melted *o*-cresol (4.1).

Place the tube in the apparatus (5.5) and allow the mixture to crystallize by cooling, stirring with the agitator (5.7).

When crystallization takes place, there is a small increase in temperature. Note the maximum value obtained, t_1.

7.3 Determination

Remelt the mixture, at a temperature not exceeding t_1 by more than 5 °C, using the water bath (5.6). Place the test tube (5.3) into the apparatus (5.5) maintained at a temperature 5 °C below t_1, checking with the ordinary thermometer (5.2).

When crystallization begins, or when the temperature of the mixture has fallen to a value 3 °C below t_1, stir continuously by means of the agitator (5.7). Take care that the bulb of the thermometer is always completely immersed. Induce the crystallization by rubbing the wall of the test tube with the bulb of the thermometer. Note the maximum temperature at which the mixture crystallizes, t_2.

Repeat the determination until the two highest values obtained for t_2 do not differ by more than 0,2 °C.

If supercooling occurs, induce the crystallization by adding a small crystal of the 1,8-cineole-*o*-cresol complex (4.3).

If t_2 is below 27,4 °C, repeat the determination after the addition of 5,10 g of the 1,8-cineole-*o*-cresol complex (4.3).

8 Expression of results

The content of 1,8-cineole, corresponding to the highest temperature observed (t_2), is given in the table.

If 5,10 g of the 1,8-cineole-*o*-cresol complex (4.3) has been added, the 1,8-cineole content of the sample, expressed as a percentage by mass, is given by the formula.

$$2\,(A - 50)$$

where A is the percentage of 1,8-cineole indicated in the table.

The results shall be expressed to two significant figures. The content of 1,8-cineole, corresponding to the highest temperature observed (t_2), is obtained, where necessary, by interpolation from the data in the table.

9 Test report

The test report shall state the method used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that might have affected the results.

The test report shall include all details required for the complete identification of the sample.

Table — 1,8-Cineole content, as a percentage by mass, as a function of the crystallization temperature of the essential oil-*o*-cresol mixture

Crystallization temperature	1,8-Cineole content	Crystallization temperature	1,8-Cineole content	Crystallization temperature	1,8-Cineole content	Crystallization temperature	1,8-Cineole content
°C	% (*m*/*m*)	°C	% (*m*/*m*)	°C	% (*m*/*m*)	°C	% (*m*/*m*)
24	45,5	32	56	40	67	48	82
25	47	33	57	41	68,5	49	84
26	48,5	34	58,5	42	70,5	50	86
27	49,5	35	60	43	72,5	51	88,5
28	50,5	36	61	44	74	52	91
29	52	37	62,5	45	76	53	93,5
30	53,5	38	63,5	46	78	54	96
31	54,5	39	65	47	80	55	99

Approximate dimensions in millimetres

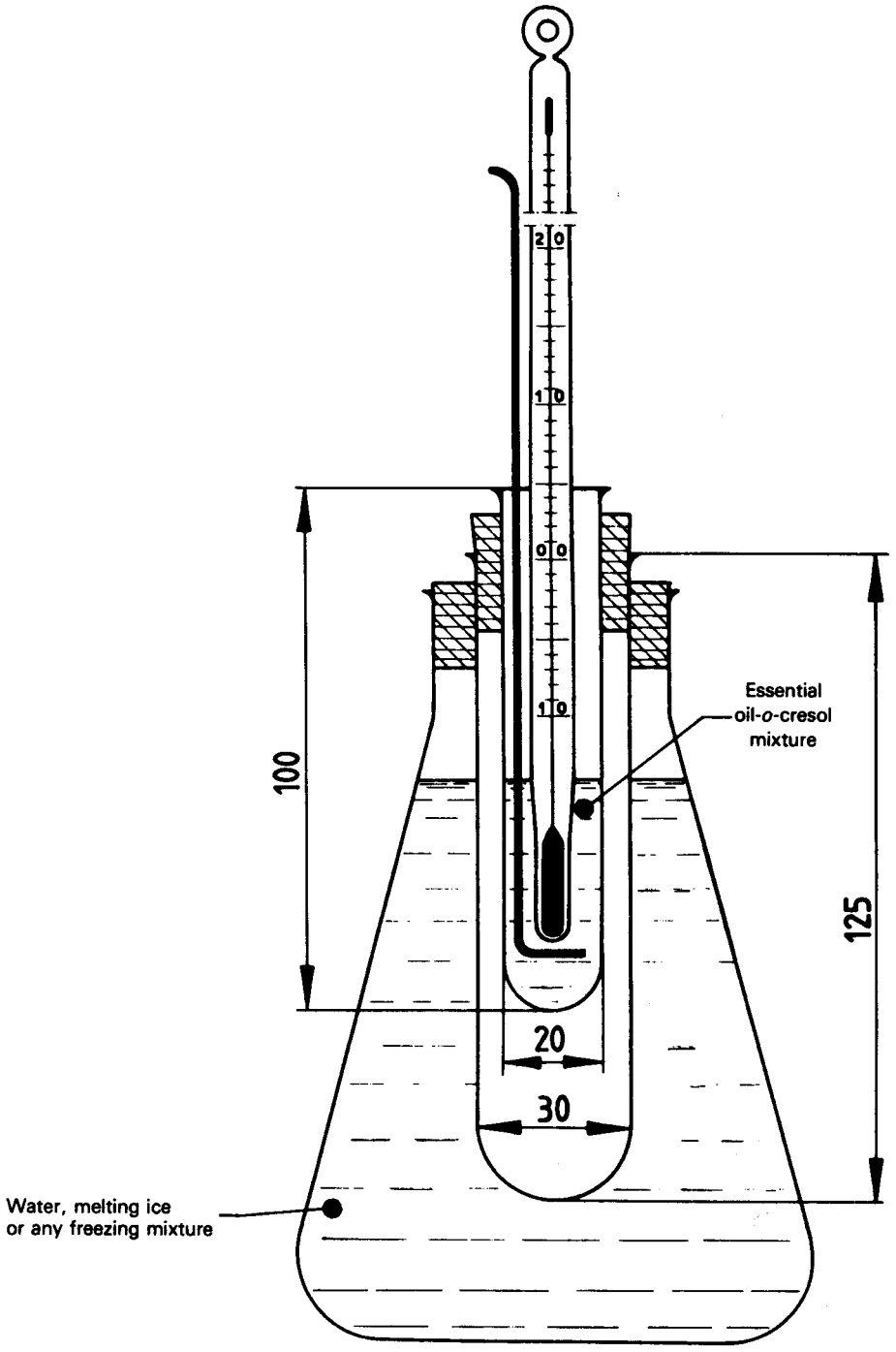

Figure — Example of apparatus assembly for determination of freezing point

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc : No. **PCD 18 (2207).**

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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